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"Methods for Purifying the Inert Gases"

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METHODS FOR PURIFYING THE INERT GASES*[Figures are in the Appendix]*

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[Author's Abstract]

This report describes a method for ridding inert gases of active impurities which is accomplished both in special clean tubes and directly in tubes already unsoldered. The method is to introduce, into the space to be purified, some metallic sodium by electrolysis of a glass ball and to treat after this the gas mixture to discharges in a high-frequency field. The description of the purification methods applied in the case of various impurities is accompanied by experimental data on the results obtained.

[Text]

Discharge tubes filled with inert gases find themselves employed more and more with each year in technology and various scientific-research works. The purity of the gases filling these tubes completely governs the stable behavior of the tube, its discharge voltage stability of the falling characteristic, and distribution of energy radiated by the lit tube. In the latter case, where the tube operates as a radiator in a definite region of the spectrum, the purity of the gas possesses decisive significance. While

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making numerous specially designed gas tubes, the laboratory of the Electro-Physical section of GPTI (State Physico-Technical Institute) solved some problems concerning the purification of gases. In the process of this work, the laboratory introduced some changes in former methods which the present articles will describe.

It is relatively simple to purify helium which is satisfactorily purified by passing over activated coal at temperatures of liquid air. For other inert gases this method is absolutely inapplicable, since their absorption coefficients lie in the same limits as those for activated gases, as is obvious from the attached table of Dewar (1914):

One gram of carbon at temperatures of liquid air absorbs

Ne	...	15 cm <sup>3</sup>	N <sub>2</sub>	...	155 cm <sup>3</sup>
Ar	...	175 cm <sup>3</sup>	H <sub>2</sub>	...	135 cm <sup>3</sup>
O <sub>2</sub>	...	230 cm <sup>3</sup>	Air	...	180-200 cm <sup>3</sup>

The absorption coefficient of neon under same conditions is significantly higher than that of helium, because of which helium can be separated from other inert gases.

The classical methods of purification by various absorbers in most cases are seldom applied in vacuum technology directly. Apparatus working on this principle generally consist of a certain closed ring through which circulates the gas to be purified, passing across a system of suitable absorbers. Since the stopcocks in the ring connecting tubes, and other general parts of the apparatus cannot of necessity be completely degasified by heating, the

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purification is doubtful and hence this method is not applicable in every case for the obtaining of spectrally-clean gases. The great advantage of these methods is that very great quantities of strongly polluted materials can be purified, but the product obtained seldom satisfies directly the strict requirements of present-day vacuum technology without further purification. The method widely used at present for purifying magnesium by diffusion is ideal for removing such impurities as nitrogen, but the complete removal of hydrogen and water vapor is doubtful. It is better to refer to the usual absorption of hydrogen as "the fastening" of it to the container walls by magnesium particles rather than as "chemical assimilation", since the structure of the hydrogen magnesium compound is not completely established.

The applicability, therefore, of such a method of purification in the case of strong pollution by hydrogen and water vapor becomes clear. The enormous advantage here in comparison with other methods is the possibility of directly de-polluting the metal in the still unsoldered tube, rather than excluding all connecting tubes unavoidably serving as sources of impurities. The only shortcoming of the method, and this only in the case of a particularly exact dose of gas, is the lowering of its gas pressure after de-pollution of the gas molecules "fastened" to the bulb walls of the de-polluted metal. The loss of gas quantitatively escapes the control of the experimenter, and the question of the final gas pressure in the tube remains uncertain.

\* [Note: "Bulb" here means "gas cylinder"]

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Most flexible and universal is the method for purifying inert gases by means of alkali metals during ionization by a current at fixed temperature. In this connection a number of works have appeared on the absorption of active gases by sodium, calcium and their alloys serving as electrodes of the discharge tube. Salet was the first to discover the absorption of nitrogen by sodium during discharge of a Geissler tube. The manometer connected to the tube, in this experiment, showed a gradual decrease of pressure to the value corresponding to that indicating a complete vanishing of free nitrogen. Warburg introduced by electrolysis water glass (sodium silicate) into the discharge tube, and thus successfully absorbed with it the impurities hydrogen and water vapor without noticeable combination of nitrogen. The contradictions between Zehnder's two experiments are explained by the bulb's high temperature at which sodium nitrogen compounds dissociate, or by the fact that the sodium deposited on the glass over the electrolyte does not appear as the electrode. The validity of both assumptions has been experimentally verified in the works described below of the GFTI laboratory. Zehnder succeeded by electrolysis in obtaining a combination of sodium with nitrogen. Having a much lower temperature, the nitrogen combined on the surface of the sodium sublimite on the cathode, but not on the anode immersed in the amalgam, whose temperature was of the order of  $300^{\circ}\text{C}$ . The author suggests that the compound formed corresponds to the formula  $\text{Na}_3\text{N}$ .

The absorption of active gases by alkali metals was investigated most extensively by Gehlhoff. The observations were carried out in an apparatus, in which solid calcium or sodium or their liquid alloy introduced under a

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vacuum served as the absorbing electrode. The general form of the apparatus and the disposition of electrodes are clearly shown in the attached diagram in which the alkali metal serving as the electrode is cross-hatched. A nearly similar work was carried out by Mey considerably earlier, which established that under these same conditions of discharge, the calcium-sodium alloy unites significantly better with nitrogen than does solid calcium and, particularly, sodium. The absorption of nitrogen occurring under these conditions is directly proportional to the quantity of electricity passing through the apparatus. He verified the phenomenon noticed by Zennder; namely, that an alkali metal when appearing as a cathode absorbs gas more actively than when the current flows in the opposite direction. Thus with current strength of 1 mA flowing 1 minute, the alloy-cathode absorbs 0.000033 gram of nitrogen, but absorbs approximately 10 times less when serving as an anode. In the latter case, Gowdry also noticed the distribution of the hydride formed earlier and explained this by the action of cathode rays on it. When analyzed, the films formed on the alloy during discharge proved to be the compound  $K_3N$  and partly  $Na_3N$ . By the method described, Mey removed the impurities oxygen, nitrogen, hydrogen and water vapor, thus obtaining spectrally pure helium as the final result. Under these same conditions, Dember obtained uniform quality of helium and argon. In investigations of cesium and rubidium vapors, Gehlhoff and Fotgart found that these vapors, at definite temperatures and under influence of the glowing discharge, very quickly and cleanly unite with the hydrogen, carbon monoxide, oxygen and nitrogen so that the residues of these gases cannot be detected even by spectroscopy. The absorption of hydrogen, as their data

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showed, proceeds best at temperature 290°; sodium, 175°; calcium, 120°; for rubidium, 120°; and cesium, 1000°C. Nitrogen is quickly and cleanly absorbed at considerably much lower temperatures. The absorption of the inert gases under these conditions was not given the stated method being used for removing them of their residues. In contrast to the above-mentioned report of Dember, Hotgart verifies the impossibility of one's obtaining argon free from hydrogen with greater than 98 percent content of argon when using exclusively the method of discharge over sodium-calcium alloy.

The works of the various authors cited above sketch sufficiently clearly the possibility of using alkali metals as absorbers of active gases. The purification process itself, however, as formulated by Mey, Hotgart and Gehlhoff, is but little applicable immediately in practice. The contradictory data on the combination of nitrogen with hydrogen raises doubts as to their complete absorption under all conditions of purification. Another shortcoming is the necessity of a special purifier and a series of connecting tubes for efficiency. Mey's method, as the works in the laboratory showed, give good results if the gas is investigated immediately after purification in the same container.

During its transfer from the purifier to the apparatus, the gas is unavoidably polluted. Its passage through the connecting tubes is accompanied by the capture of (a) gases adsorbed on the glass and (b) vapors of mercury and lubricants. Even if the latter vapors are successfully condensed partly in the liquid-air trap specially set up then the former vapors pollute the

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apparatus supposedly filled with pure gas. All things considered, the pollutions occur even during unsoldering itself of the apparatus because of the gases evolved from the highly heated glass. This naturally gives rise to the desire that the gas in the already unsoldered lamp can be purified or that the gases liberated during the course of prolonged operations with the tubes can be absorbed.

During practical work with gas lamps, for the purpose of eliminating the above-enumerated shortcomings of the various methods, certain changes were introduced in the procedure for refining gases by alkali metals, which changes gave in a number of cases positive results and great confidence in the purity of the final products. Sodium was introduced into the purifier or directly into the lamp bulb by electrolysis of glass. This procedure ensures an absolutely pure alkali metal that is devoid of oxides incrustations, lubricant and adsorbed gases. Electrolysis itself is realized in the following manner (See Figure 2): The electrolyte anode consists of a fused mixture of equi-molar quantities of calcium and sodium nitrates at temperatures of the order of 260°C. At higher temperatures the glass in the presence of metallic sodium becomes yellow but in some cases devitrification leading to damage of the apparatus occurs. The latter phenomenon, moreover, always occurs if pure sodium nitrate is used as the electrolyte. Obviously it amounts to utilizing varieties of ordinary sodium silicate, subjected to electrolysis. A half-period rectifier with voltage from 0 to 6000 V, regulated by potentiometer  $\pi$  in the primary winding of transformer T

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(See Figure 2) serves as the source of current. Consequently, in the high-voltage circuit a supplementary resistance  $R$  of the order of 100,000 ohms is included. The voltage required for the initial discharge and necessary current strength depend mainly on: gas pressure, form of container, and distance from cathode to surface of glass immersed in hot bath, and temperature of bath itself. The current strength during electrolysis depends on the area of the glass immersed in the electrolyte, and equals 0.5 to 1 mA/cm<sup>2</sup>. During electrolysis, a great part of the decrease in voltage occurs in the supplementary resistance  $R$  and in the glass. The decrease in voltage is insignificant in the gas ionized in the tube because of the falling characteristic of gaseous discharge. Because of the considerable power released in it the glass suffers an increase of temperature and the current in the beginning of electrolysis increases, while the temperature of the glass and consequently its resistance do not come into equilibrium. This occurs mainly at low temperatures of the bath and high voltages. As experiments showed, the walls of the glass immersed in the bath ought to be, if possible, of the same thickness; otherwise the glass is frequently punctured, which can possibly be explained by the fact that the more current passing through the thin places of the bulb of less resistance causes local overheating, so that further increase in current leads to a rapid destruction of the glass.

During electrolysis such admixtures of active gases and sodium compounds unite as do not disassociate at the given temperature; for example, O<sub>2</sub>, Cl<sub>2</sub>, CO<sub>2</sub>, CO and S. Such admixtures as N<sub>2</sub> and H<sub>2</sub> dissociate at sufficiently great temperatures of electrolysis, and hence complete absorption cannot be

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accomplished. Control of the process by spectroscopy shows the rapid vanishing of all gases except the latter gases, the spectrum of which is independent of how long electrolysis continued. This phenomenon can be explained as a result of established dynamic equilibrium between absorbed and liberated gases, due to dissociation. In order to displace the equilibrium constant, the temperature of the mixture in the discharge must be lowered. To do this, as experiments show, it is best to ionize the gas by way of capacitances with high-frequency current at room temperature. Under these conditions, compounds of sodium with hydrogen and nitrogen are more stable. This can be explained by the decreased dissociation at given temperatures or other improved conditions surrounding the forming chemical compounds. Mercury vapors in these quantities that are met in work with mercury vacuums without liquid-air condensation do not require a special consideration, since free sodium in the apparatus completely absorbs mercury.

Ionization by high-frequency currents was carried out by us by means of a 200-watt tube oscillator with a frequency of the order of 10,000 kilocycles (See Figure 3). The light obtained by this bears a somewhat different character than ordinary electrode discharge with direct or alternating current. At present, as in recent times, ordinary applications of pressure give a concentrated light in a small space, but high-frequency ionization gives a diffused light that spreads over the whole space of the container. The prepared tubes are ionized by simply moving them closer to the covering of condenser C (See Figure 3) connected to the anodes of oscillator tubes.

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If, at great gas pressures (10-20 mm), ionization does not take place, then ionization can be brought about by rotation of the bulb around a precipitated sodium foil strip and by direct connection of it to the anode of the oscillator.

At pressures of the order of 20-50 mm Hg, which occur in purifiers soldered to vacuum-producing devices, we need intake like that illustrated on Figure 3. High-frequency oscillations induced in induction coil L are on one side on the electrode of purifier A and on the other side on coil band K wound around the sodium precipitated on the glass. Ionization by arbitrary means of obtaining it should not be so strong that overheating occurs in the tubes with the gas to be purified. In the correct case, ionization is regulated by varying the coefficient of mutual induction between induction coils L and L'. The sodium settling on the vessel's walls is an electrode having large absorbing surface and alternating in sign. A large effective surface of sodium decreases the current strength per unit area, and thus the apparatus remains at a comparatively low temperature, which makes possible a better unification of the hydrogen and nitrogen.

Such a method for purifying gas in a high-frequency field can be considered sufficient, in most practical cases, freeing inert gases of all active mixtures. The behavior here of hydrogen and nitrogen deserves somewhat closer attention, which (H and N) we will discuss now in greater detail. During preparations of special photographic tubes rich in ultraviolet rays, we employed mixtures of argon with nitrogen, as received directly from dried air. The nitrogen is cleanly and quickly freed of oxygen and other

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admixtures present in the atmosphere of the laboratory by simple electrolysis; (as the experiment following below will show) the nitrogen is brought into discharge in an interesting manner. The special purifier, as in similar experiment of Lalet, is connected to a U-shaped manometer, with open end soldered onto the vacuum producing device and then evacuated at a temperature of  $450^{\circ}$  to a pressure of  $10^{-6}$  mm (See Figure 4). The purifier, filled afterwards with a mixture of dry air and some inert gas, is unsoldered from the vacuum device.

In one particular case, the gas consisted of a mixture of helium and air under ordinary pressure of 14 mm; partial pressures were: helium 4 mm (28.6%), nitrogen 8 mm (57.1%), and oxygen 2 mm (14.3%). The part A of the purifier was immersed in a hot bath, as was pointed out earlier, and sodium introduced electrically. With the dark blue appearance of the discharge on the glass walls contiguous to the bath container the glass became covered with a dark blue light, which represents the spectrum of air without indications of helium and sodium. After some time, the blue light passes rather sharply over into yellow, which, in agreement with Harburg's observation, signifies the complete absorption of oxygen by sodium. This moment corresponds to the appearance in nitrogen's characteristic spectrum, of a clear sodium doublet. Here electrolysis was interrupted, and the pressure in the purifier cooling to room temperature turned out to be 12 mm. Both spectrum and remaining pressure testify to the complete absence of free oxygen; also the quantity of sodium introduced completely coincides with the amount of

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oxygen required for absorption as calculated by the stoichiometric formula  $\text{Na}_2\text{O}$ . Sodium oxide covered the entire bulb on that part of the container immersed in the bath with a white semi-transparent thin deposit. In the presence of free oxygen, sodium readily oxidizes with the simultaneous appearance of a powder on the glass surface. With repeated electrolysis, sodium was introduced into the purifier in significantly larger quantities than required for the chemical composition corresponding to  $\text{Na}_3\text{N}$ . After cooling of the purifier, no decrease in pressure was noticed; obviously, the temperature of electrolysis is not associated with noticeable quantities of nitrogen combined with sodium. With further ionization, the pressure, as read on the manometer, in the purifier held in a high-frequency field begins to decrease, and sodium combined with nitrogen covers the surface with a brownish deposit. The spectroscopic picture shows a rapid weakening of the intensity of the nitrogen spectrum and gradual appearance of helium lines in its background, the intensity of which increases noticeably. After some time, the nitrogen spectrum vanishes without trace and only mercury lines disturb the purity of the helium spectrum, with mercury vapors continuously rising from the manometer. Finally, the gas pressure in the container becomes 4 mm, which corresponds to the remaining helium. When complete absorption of nitrogen was verified spectroscopically, the purifier was placed in an electric oven and then gradually heated up to a temperature of  $350^\circ\text{C}$ . The pressure in the container was determined after every  $5^\circ$ , according to which the greatest yield of nitrogen occurred at around  $275^\circ\text{C}$ .

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The overall pressure after heating of the purifier corresponded to the previously given pressure of helium and nitrogen. A second ionization of the mixture again gave a helium spectrum free from nitrogen. The agreement of the temperature ( $275^\circ$ ) of decomposition of the nitrogen-sodium compound with the data of de-our, Clausius and Lecher (12) on the decomposition of sodium azide ( $\text{Na N}_3$ ) indicated indirectly that in the case of high-frequency discharge a transformation of that compound ( $\text{Na N}_3$ ) but not that ( $\text{NaN}_3$ ) obtained by Ray<sup>13</sup> by a simple discharge can occur.

The most difficult of all to remove no matter what the methods, is a hydrogen admixture. Even if pollution by pure hydrogen is not initially encountered so frequently as the original admixture in the gas, still traces of hydrogen often appear afterwards as a result of decomposition by sodium of such initial pollutions as  $\text{PH}_3$ ,  $\text{H}_2\text{S}$ ,  $\text{HCl}$ ,  $\text{CH}_4$ ,  $\text{C}_2\text{H}_2$ , water vapors, and vacuum sealing compounds.

The behavior of hydrogen in discharges over alkali metals is more definite, since there is a greater literature on hydrogen compounds of sodium. A whole series of investigators obtained sodium hydride by heating the metal in hydrogen. The optimum temperature for the formation of a compound proves to be  $350^\circ\text{C}$ ; and the temperature of dissociation with the greatest yield of hydrogen, according to Moissan is  $360^\circ\text{C}$ . Sodium hydride is attributed a constant composition corresponding to the definite chemical composition chemical composition  $\text{NaH}$ . The pressure-temperature curve of hydrogen during dissociation of hydride is represented approximately as in Figure 5. The

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graph curve can rises all data points by (Troost and Bouteville) Keyes, Zerkov and (Huttig and Trodkorb). Data of the latter authors deserve particular attention since they refer partially also to room temperature. One must notice here two details pointed out by them: 1) hydrogen's pressure on sodium hydride does not attain a constant magnitude and, after the original rapid increase, gradually increases almost in direct proportion to time; 2) the final pressure depends on the amount of hydrogen absorbed (for instance, 1 gram-atom of sodium absorbing 0.9064 gram-atom of hydrogen gave after 6 hours a pressure of 2.969 mm, in which time the very same quantity of sodium absorbing 0.736 gram-atom of hydrogen after 29 hours showed a pressure of only 1.010 mm). Cowdry gives analogous indications, having studied the absorption of hydrogen by a calcium-sodium alloy in discharge.

The short survey above shows that hydrogen, requires the same purification methods as nitrogen, with only this difference: if there are large quantities of absorbed hydrogen then it is desirable to isolate the hydride formed, in order that the hydride's dissociation occurring with the passage of time does not vitiate the results. In the electrolysis process, despite favorable conditions, hydrogen is completely captured by sodium only when hydrogen is present in traces. But when there are large quantities of hydrogen, the high-frequency method of treating the gas is absolutely necessary. The formation of hydride at low temperature of discharge does not contradict at all the conditions of formation introduced earlier. Cowdry explains the ability of sodium to combine with  $H_2$  at low temperatures by local overheating of the surface at points where the ions strike. The calculations by him

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show that the charged particle, possessing a velocity of 10 V, gives a sufficient amount of energy to increase the temperature of 10 atoms by 400°C. The formation of hydride ought to occur in this favorable zone.

In the preparation of Plucker tubes, we had to remove large quantities of hydrogen. For the removal of the hydride formed, the tubes were supplied with an appendage A (see Figure 6), which served for the introduction sodium and which was unsoldered after final purification. For the purpose of studying the conditions governing the absorption of hydrogen filling the tube the hydrogen was collated with sodium from 10 to 60%. At the beginning of electrolysis, in the case of large hydrogen pressures, the spectroscopist gives a strong hydrogen spectrum with a scarcely noticeable yellow helium line (5875.96 and 5875.021). In the course of time, the reddish-violet hydrogen lumination rather sharply gives way to a white helium lumination. In the spectroscopist a bright helium spectrum appears simultaneously with the gradual diminishing of the hydrogen lines, but the hydrogen lines never vanish completely with further electrolysis. After high-frequency ionization of the gas in the the hydrogen spectrum can be noticed, in the case where there are large quantities of the gas, only in spectrograms with large exposures and vanishes completely if the hydrogen is present in quantities less than several percent. At the same time, the hydrogen lines inevitably appear in the course of time if the bulb A, serving for purification, is not unsoldered. These observations completely verify the data of Huttig and Brodtkorb concerning the instability of sodium hydride at room temperature. As it follows from the data introduced to obtain inert gases free from hydrogen requires that the concentration of hydrogen be lowered as much

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as it is possible by passing through some chemical absorber (for example, over  $\text{CuO}$  at high temperature and  $\text{P}_2\text{O}_5$ ), and that the purification with alkali metals in discharge be left only for removing traces of hydrogen.

Inert gases are absorbed significantly more rapidly when the sodium is spread out as a thin layer on the large glass surface since the corresponding sodium compounds of hydrogen and nitrogen are formed mainly on the surface, not penetrating deeply into the depths of the metal. Thus it is more suitable to carry out electrolysis in large-diameter purifiers and under less gas pressures in the apparatus. From time to time it is necessary to clean the container from previously absorbed gases by heating it to a temperature of around  $400^\circ\text{C}$  and subsequently exhausting. As experiments show, moderate electrolysis can in the course of several years systematically get rid of compounds that accumulate in the container. The supplementary bulb-purifier described above (Figure 6) successfully isolates the products of purification and the residues of metallic sodium from the working parts of the apparatus. In the case where the apparatus can not be sufficiently well degassed and one can anticipate in the process of operation the introduction of pollution from the electrodes and glass, it is reasonable to carry out purification in the apparatus itself by leaving certain quantity of sodium behind for repeated operations. In this case, however, one must always take care that the sodium and its compounds be localized in the part of the apparatus not subject to heating and be removed from the discharge action.

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The above-described method of purification of inert gases by electrolytic sodium in a high-frequency field has been extensively for several years employed in the GFTI Laboratory. (Note: GFTI: State Physico-technical Institute). In practice, the Laboratory has purified argon, neon, and helium from the atmosphere and has removed those pollutions inevitably present under laboratory conditions. The following mixtures, sometimes in quantities up to 200-300%, were introduced artificially: air,  $O_2$ ,  $N_2$ ,  $H_2O$ ,  $H_2S$ ,  $HCl$ ,  $CO_2$ ,  $CO$ ,  $SO_2$ ,  $Hg$  and others. Spectroscopic investigation of the products of purification generally gave satisfactory results except for some doubtful cases where the negative results could be explained perhaps by external causes such as insufficient original degasification of the apparatus or leakage. In most cases, it is easy to follow visually the course of purification without use of the spectroscopy by the abrupt variations in the character of lumination. The greatest visual effect is obtained in the purification of neon lamps polluted by air. When glowing from direct or alternating current, seemingly relatively pure, the lumination of neon at high-frequency ionization suddenly changes. The smallest admixtures of impurities emit a bluish-violet hue, frequently suppressing completely the original orange light of the inert gas. In the presence of electrolytic sodium, high-frequency ionization quickly suppresses the parasitic lumination, originally dominant, which in the course of several seconds gives place to the clear light of neon. This change in lumination is quite characteristic and starts so suddenly that the phenomenon can serve as a visual

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visual criterion for detecting the end of purification. The table below contains data on the purification of neon lamps that have been rejected because of a bluish shade when they glow and under high-discharge voltages.

The figures in the table picture the lowering of the discharge voltage and the stable behavior of the lamp with time;  $V_1$  is the discharge voltage before purification,  $V_2$  is that after ionization in the presence of electrolytic sodium,  $V_3$  is the voltage over 11 months after purification, and  $V_4$  is the voltage after secondary electrolysis and ionization. The discharge voltage was measured by use of a 50-cycle current. After an interval of 11 months, the lamps were found in almost continuous operation; a scarcely noticeable bluish hue in the lumination appeared in them, thus indicating little concerning the magnitude of the discharge voltage. Lamp No. 110 was not cleared completely since it had been filled with sufficiently pure gas.

No.	$V_1$	$V_2$	$V_3$	$V_4$	No.	$V_1$	$V_2$	$V_3$	$V_4$
62	150	105	100	100	111	220	83	80	80
68	115	80	80	80	112	250	85	80	80
69	290	90	103	80	113	150	105	90	108
107	290	115	110	102	116	240	80	80	80
110	180	-	200	-	-	-	-	-	-

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The most characteristic sign of the purity of the filling gas is lowering of the discharge voltage. For comparatively large pressures of neon, the discharge voltage had to be lowered to 75 V of alternating current; and for lesser pressures of argon and helium, to 60 V. The stability of the discharge voltage serves as another criterion of purity; namely, consisting in the absence of so-called "polarization" of electrodes (i.e., in the absence of a difference between two successive values of the discharge tension). Where two successive determinations of discharge voltage sometimes differ by 15-20 V in an unpurified lamp, the lamps cleaned by the above-described method show a fluctuation of 1-2 V. Washing the electrodes with sodium in electrolysis possesses especially great significance in the sense of decreasing "polarization" in them. Such a treatment changes somewhat the surface's external appearance and sometimes makes it noticeably photoelectric. After electrolysis, naturally, the sodium must pass with repeated heating of electrodes and glass over into the lamp's non-operating heated portion.

The various practical results briefly mentioned above convince us that the purification method by electrolytic sodium in high-frequency discharges with complete knowledge of the purification processes can be utilized more extensively than merely in purely laboratory practice. It is still premature to report present exact numerical data concerning the behavior of absorption and the stoichiometric forms of the sodium compounds prepared,

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since the work is not completely finished and this article is of the nature of a preliminary report. The spectrograms obtained during this work of recording the various stages in the process of purification as well as data of chemical analysis of the compounds formed will be reported more fully later on.

In conclusion, I consider it my obligation to express gratitude to Professor A. A. Chernyshev and Engineer A. E. Moskvina for their courteous attention and assistance in this work.

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## APPENDIX: FIGURES

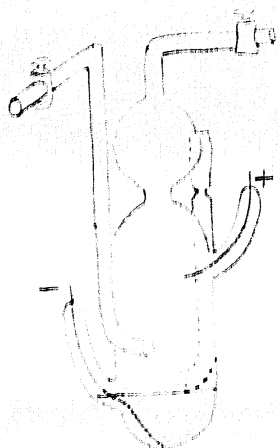


Figure 1.

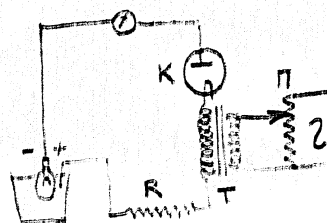


Figure 2

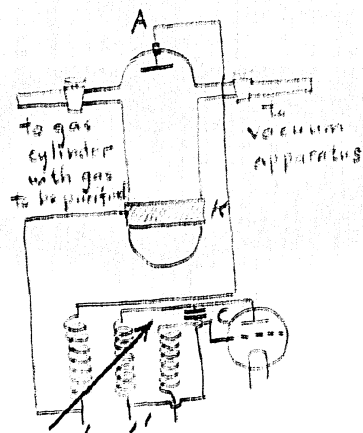


Figure 3

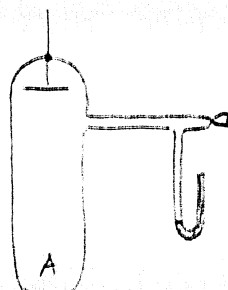


Figure 4.

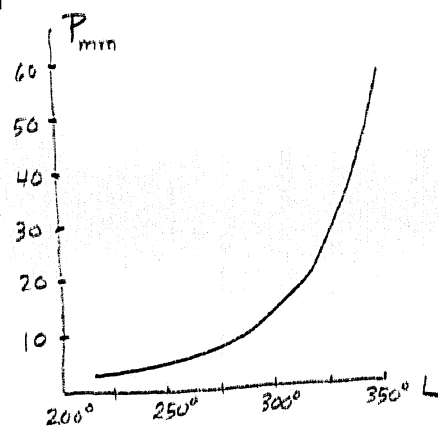


Figure 5.

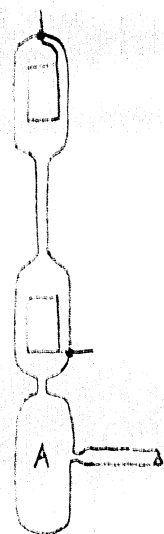


Figure 6.

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